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A Variable Temperature X- and W-Band EPR Study of Fe-Doped SiCN Ceramics Annealed at 1000, 1100, and 1285 °C: Dangling Bonds, Ferromagnetism and Superparamagnetism

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Abstract Polymer-derived SiCN ceramics, annealed (also referred to as pyrolyzed) at 1000, 1100, and 1285 °C, and doped with Fe(III) acetylacetonate, are investigated by electron paramagnetic resonance (EPR) from 4 to 120 K at X-band (9.425 GHz). In addition, the SiCN ceramic, annealed at 1100 °C, was studied by EPR at 300 K at W-band (93.96 GHz). There was observed a significant increase in EPR linewidth due to dangling bonds ($g = 2.001$) below 20 K at X-band. The low-field X-band FMR line ($g \approx 12$) indicated the presence of ferromagnetic Fe_5Si_3 crystallites. There were found two EPR lines due to carbon-related dangling bonds: (1) those present as defects on the surface of the free-carbon phase (as sp^2 carbon-related dangling bonds with $g = 2.0011$) and (2) those present within the bulk of carbon phase (as sp^3 carbon-related dangling bonds with $g = 2.0033$). On the other hand, the intense low-field EPR signal observed at X-band was not observed at W-band. As well, there was observed splitting of the single broad EPR signal observed at $g = 2.05$ at X-band into two signals at W-band at $g = 1.99$ and $g = 2.06$, due to two different Fe-containing superparamagnetic nanocrystallites. Two new EPR signals, not observed at X-band, were observed at W-band, namely at $g = 2.28$ and $g = 3.00$, which are also due to $g_{\|}$ of these superparamagnetic nanocrystallites.

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